भारतीय मानक
Indian Standard

IS 1448 (Part 50) : 2021

पैट्रोलियम और इसके उत्पादों के परीक्षण की विधियाँ

भाग 50 नई एवं प्रयुक्त स्नेहकों में क्लोरिन (सोडियम एल्कोहोलेट पद्धति)

(तीसरा पुनरीक्षण)

Methods of Test for Petroleum and its Products

Part 50 Chlorine in New and Used Lubricants (Sodium Alcoholate Method)

(Third Revision)

ICS 75.080, 75.100

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June 2021 **Price Group 3**

Methods of Sampling and Test for Petroleum, its Products, Gaseous Fuels and Lubricants Sectional Committee, PCD 01

FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards on after the draft finalized by the Methods of Sampling and Test for Petroleum, its Products, Gaseous Fuels and Lubricants Sectional Committee and approval of the Petroleum, Coal and Related Products Division Council.

This standard was revised for the second time in 1977 and was adapted with permission from the joint publication ASTM D 1317-64/ IP 118/65.

In this third revision, reference clause has been included.

The composition of the Committee responsible for the formulation of this standard is given in Annex A.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (*revised*)’.

Indian Standard

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

PART 50 CHLORINE IN NEW AND USED LUBRICANTS (SODIUM ALCOHOLATE METHOD)

(Third Revision)

1 SCOPE

This Indian Standard (Part 50) describes a test procedure for the determination of chlorine content of lubricating oils and greases with which chlorinated additives have been blended. It is equally applicable to new or used oils and greases and to the additive concentrate. The procedure assumes that compounds containing halogens other than chlorine will not be present.

2 REFERENCES

The following Indian Standards contain provisions which through reference in this text constitute the provisions of the standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
361 : 2009	Normal butyl alcohol, technical — Specification (*third revision*)
1070 : 1992	Reagent grade water — Specification (*third revision*)
1447 (Part 1) : 2021	Methods of sampling of petroleum and its products: Part 1 Manual sampling (*second revision*)
1447 (Part 3) : 2021	Methods of sampling of petroleum and its products: Part 3 Method of sampling of semi-solid and solid petroleum products (*second revision*)
1745 : 2018	Petroleum hydrocarbon solvents — Specification (*third revision*)

3 OUTLINE OF THE METHOD

The sample, dissolved in a low boiling hydrocarbon mixture, is boiled under reflux with metallic sodium and *n*-butyl alcohol. Under these conditions, the chlorine is converted to sodium chloride which is then extracted with water. The chloride in the extract is determined volumetrically by titration with silver nitrate in the presence of thiocyanate.

4 APPARATUS

4.1 Reaction Vessel — Consisting of a 300 ml round-bottom flask with standard-taper joint and surmounted with a water-cooled condenser (*see* Note).

NOTE — The condenser tube shall be wide enough to allow rapid reflux without hold-up.

5 REAGENTS

5.1 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

5.2 Benzyl Alcohol — The benzyl alcohol used shall not show a visible precipitate when tested with alcoholic silver nitrate ($AgNO_3$) solution.

5.3 *n*-Butyl Alcohol — *see* IS 361.

5.4 Ferric Alum Indicator Solution — Dissolve 350 g of ferric alum $Fe_2(SO_4)_3.(NH_4)_2SO_4.\ 24H_2O$ in 1 litre of water and add 200 ml of aerated nitric acid (1 : 2).

5.5 Iso-amyl Alcohol — The iso-amyl alcohol used shall not show a visible precipitate when tested with alcoholic silver nitrate ($AgNO_3$) solution.

5.6 Naphtha — Paraffinic, distilling between 99 and 121 °C.

(*or*)

5.7 Petroleum Hydrocarbon Solvent (100/120) — *see* IS 1745.

5.8 Nitric Acid (1 : 7) — Mix 1 volume of concentrated nitric acid (relative density 1.42) with 7 volumes of water. Aerate to remove oxides of nitrogen.

5.9 Phenolphthalein Indicator Solution (10 g/l) — Dissolve 1 g of phenolphthalein in iso-propyl alcohol (60 percent) and dilute to 100 ml with the alcohol.

5.10 Standard Silver Nitrate Solution (0.1 N) — Dissolve 16.9890 g of dry (1 h at 110 °C) silver nitrate ($AgNO_3$) in water and dilute to 1 litre in a volumetric flask with water.

5.11 Sodium Metal

5.12 Sodium Hydroxide Solution (80 g/l) — Dissolve 80 g of sodium hydroxide in water and dilute to 1 litre.

5.13 Standard Thiocyanate Solution (0.05 N) — Dissolve 3.80 g of ammonium thiocyanate (NH_4CNS) or 4.85 g of potassium thiocyanate (KCNS) crystals in water and dilute to 1 litre. To standardize this solution, measure 40 ml into a flask containing 5 ml of nitric acid (1 : 7) and 50 to 60 ml of water. Swirling the flask constantly, measure 22 to 24 ml of 0.1 N silver nitrate solution from a burette; then stopper and shake for 20 to 30 s. Add 2 to 3 ml of ferric alum indicator solution and titrate with the thiocyanate solution to a red end point. Calculate the normality of the thiocyanate solution as follows:

$$\text{Normality of thiocyanate solution} = \frac{A \times N}{B}$$

where

A = volume of standard silver nitrate solution used, in ml;

N = normality of the silver nitrate solution; and

B = total volume of the thiocyanate solution used, in ml.

6 PROCEDURE

6.1 Obtain sample as mentioned in IS 1447 (Part 1) or IS 1447 (Part 3), as applicable. Shake the sample, if liquid, to ensure homogeneity. Weigh a portion, estimated to contain 0.05 to 0.1 g of chlorine, into the 300 ml flask. Add 25 ml of naphtha or petroleum hydrocarbon solvent until the sample is dissolved.

6.2 Reaction and Extraction

6.2.1 To the solution of the sample, add 5 ml of *n*-butyl alcohol and 2.8 ± 0.2 g of freshly cut sodium. Connect the flask to the condenser and boil the liquid vigorously under reflux for 1 h. Introduce an additional 20 ml of *n*-butyl alcohol through the top of the condenser and continue refluxing until no metallic sodium remains.

6.2.2 Add 25 ml of water to the flask through the condenser, cool to room temperature, and transfer the mixture to a 500 ml separatory funnel. Wash the flask with 50 ml of nitric acid (1 : 7) and then with 50 ml of naphtha or petroleum hydrocarbon solvent. Add these washings to the separatory funnel. Adjust the acidity of the aqueous layer until just acidic phenolphthalein, using nitric acid (1 : 7) or sodium hydroxide solution for this purpose; then add 12 to 13 ml of nitric acid (1 : 7).

6.2.3 Shake the funnel and contents, and, after settling, draw the aqueous layer into a clean 500 ml glass stoppered conical flask. Wash the original 300 ml reaction flask with 25 ml of water, transfer to the separatory funnel, wash the naphtha or petroleum hydrocarbon solvent layer which remained in the separatory funnel after separating the aqueous layer; transfer this washing to the 500 ml flask. Repeat with a second 25 ml portion of water.

6.3 Chloride Determination

6.3.1 Boil the combined acidic aqueous extracts and washings until free of hydrogen sulphide, as detected by dipping a piece of lead acetate test paper into the solution (no black discoloration). If the solution is clear, proceed with the directions in **6.3.2** and **6.3.3**; if not, filter the solution through a qualitative filter paper into another 500 ml glass-stoppered conical flask. Rinse the original flask with two 25 ml portions of water, using each portion to wash the filter paper.

6.3.2 After the solution has attained room temperature, add 10 ml of iso-amyl alcohol or benzyl alcohol (*see* Note); 3 ml of ferric alum indicator and, from a burette, 0.4 to 0.6 ml of 0.05 N thiocyanate solution. Swirling constantly, titrate with 0.1 N silver nitrate solution until the red colour is discharged; then add 2 to 5 ml in excess.

NOTE — Because of the extreme toxicity of nitrobenzene its use has been discontinued, and the use of either iso-amyl or benzyl alcohol is recommended. Both of these alcohols are relatively many times less toxic than nitrobenzene. However, care should be exercised with their use, as they are considered to have moderate toxic properties.

6.3.3 Stopper the flask tightly and shake vigorously for 15 s to coagulate and remove the precipitate from the aqueous phase (*see* caution). Without refilling the burette, titrate slowly with 0.05 N thiocyanate solution until the end point is approached, as indicated by a more slowly fading red colour. Stopper the flask, shake vigorously for 20 to 30 s, and continue the titration until one drop produces a distinct reddish coloration that does not fade upon swirling or vigorous shaking.

Caution — Care should be exercised in opening the flask after shaking. Pressure may develop, causing small amounts of the acid solution to be sprayed from the mouth of the flask as the stopper is removed.

6.4 Blank Determination

Simultaneously with each set of sample determination, make duplicate blank determinations, following exactly the procedure used for the sample determinations (except for omission of the sample). Always use identical reagents for the blank determination and for the sample determination. If the titration exceeds 0.05 ml, discard the responsible reagent.

7 CALCULATION

Calculate the total chlorine content of the sample as follows:

$$\text{Chlorine, percent by mass} = \frac{\left(AN - BN_1\right)\times 35.46}{10M}$$

where

A = volume of standard silver nitrate solution used, in ml;

N = normality of the silver nitrate solution;

B = volume of thiocyanate solution used, in ml;

N_1 = normality of the thiocyanate solution; and

M = mass of the original sample used, in g.

8 REPORTING

Report the result to the nearest 0.01 percent as the chlorine content.

9 PRECISION

Results of duplicate tests shall not differ by more than the following amounts:

Chlorine Content (percent by mass)	*Repeatability*	*Reproducibility*
0.1 to 1.0	0.03	0.04
Over 1.0	3 percent of amount present	6 percent of amount present

ANNEX A

(*Foreword*)

COMMITTEE COMPOSITION

Methods of Sampling and Test for Petroleum, its Products,
Gaseous Fuels and Lubricants Sectional Committee, PCD 01

Organization	*Representative(s)*
Indian Institute of Petroleum, Dehradun	Dr Anjan Ray (***Chairman***)
Air Headquarters, Ministry of Defence, New Delhi	Wg Cmdr S. A. Tawab Wg Cmdr D. Anandraj Roy (*Alternate*)
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Indian Oil Corporation Limited, Mumbai	Shri P. D. Dusane Shri G. K. Sharma (*Alternate*)
Lubrizol India Limited, Mumbai	Shri Sharad Tarsode

Organization	*Representative(s)*
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BIS Director General	SHRI N. K. BANSAL, SCIENTIST 'F' AND HEAD (PCD) [REPRESENTING DIRECTOR GENERAL (*Ex-officio*)]

Member Secretary

SHRIMATI NAGAMANI T.
SCIENTIST 'E' (PCD), BIS

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